DIPHENYL
AND
CHLORINATED DIPHENYL
DERIVATIVES

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NOTES ON A PLANT FOR THE MANUFACTURE OF AROCLOR NO. 1269

Aroclor No. 1269 is the product distilled from chlorinated diphenyl with a chlorine content of 69%.

A brief history of these operations would probably be in order.

The first distillations were made in small steel pots fired by gas. A satisfactory product was made, but there was much decomposition with low yield.

To improve the yield, we next constructed a unit consisting of a cast iron pot with thick sides and bottom. The inside of this pot was completely machined so that the heavy plow type agitator would sweep within 1/8" of the entire interior surface. Firing was done with gas. This arrangement increased the yield but carbon building up and adhering to the shell would very often stop the agitator. This still was soon abandoned in favor of the present type of distillation equipment which gave higher recoveries with greater ease of operation.

The present still consists of a steel pot with a launder on the inside of the shell. There is a drain from this launder to the flaker (formerly to the quenching tank). The pot has a removable cover with a vent. The firing is by gas.

The suggested plans for a new plant as shown on our Drawing #Sc-2407 proposes a tank, lagged and heated by circulated hot oil or one of the Aroclors, for receiving the batch from the chlorinators. Material from this tank would be fed to a flaker, from which an elevator would elevate to an overhead bin. The above drawing shows a bin of 50,000 lbs. capacity but this can be varied according to operating schedule.

The flaked material would flow to the still pot, which rests on a platform scale and from which the exact weight for a charge is determined. In a similar way, lime is added either from a bag or similar device.

An alternate way of charging the stills would be to run in molten crude from the heated storage tank. Since the chlorinators require 40 hours to produce 7,500 - 8,000 lbs. of crude and the stills have about the same capacity, some of the crude would be stored hot for about 40 hours. Crude 1168 and 1169 held slightly above the melting point decompose to some extent. Whether this amount of decomposition would be appreciable or not is not known.

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Conversion of the liquid crude chlorinated material into either finished product or flaked solid form as rapidly as possible seems the most advantageous procedure. After a chlorinator batch is dumped into the storage tank the flaker would be started. As rapidly as stills became available molten material would be run into them until all liquid crude was exhausted. Stills would then be charged with flaked material.

The stills filled with a batch are elevated and conveyed to a steel setting lined preferably with insulating refractories. These insulating refractories are especially adapted to such batch operations because of their low capacity for heat. Firing may be by any fuel, use that which is cheapest and most convenient.

The material distills out and is flaked on a chill wheel from which it passes to an elevator and overhead packing bin.

In these operations, off grade product comes off at both the start and at the end of a batch run. It is necessary to divert this off grade material from the chill wheel. For this purpose we use a small vessel with spout. This device deflects the off grade product and prevents it reaching the chill wheel.

At the end of the run, the pot with coke, lime, etc., is lifted from the setting and removed to an area where it can be opened and cleaned.

All construction must be tight so that a minimum of dust contacts the operators. The room housing this equipment should be well ventilated both because the stills are hot and as an extra safeguard against dust. Operators in contact with such dust may develop a severe form of dermatitis.

These operations as above described are far from ideal. The batch requires close watching to prevent off-grade products mixing with the good product at the first and last of each distillation. This splitting is entirely dependent on the operator, his care and visual observations.

Another factor complicating operations is the sudden decomposition that occurs at the end of the distillation. The gas evolved contains large amounts of acid forming vapors and also some of the Aroclor 1269 as an extremely finely divided dust which is discolored and decidedly acid. Efforts made to collect this dust and rework it have not been successful. Our operations at present are to discharge these decomposition products through an exhauster and into the atmosphere. Such a procedure in a more populous community might create a nuisance.

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A better method is desired for cleaning the coke and lime residue out of the pots. At the present time the dense, tightly, adhering mass is chipped out with bars or dislodged by pounding on the pot. The still pots scale and distort as a result of the residue and are battered by the cleaning operation. Wetting the residue has been tried but did not facilitate their removal.

Vacuum distillation has been considered but certain complications present themselves. The cleaning of such a vacuum still would be a serious problem, for if distillation were carried only to the point where the residue would run out, the recovery would be low. The material sublimes readily and any air leakage would quickly cause a stoppage by the sublimed material. Then too the hold point is very close to the boiling point under reduced pressures.

Data on vacuum distillation as done by the laboratory are as follows:-

Hold Point of Batch #192		=	241°C
Charge	556 G		
Yield	542 G	=	96%
Residue	12 G	=	4%
Total Distillation Time		=	12 mins. or 29 G/min.

Distillation Range	Actual Reading	Corrected for Stem Exposure	Pressure
1st gram (approx.)	240°C	245.5°C	8 mm.
10th " "	255	258.7	7.5 "
125 " "	255	258.8	7.5 "
275 " "	265	267.1	7.5 "
525 " "	264	268.2	7.5 "
542 " "	270	274.2	7.5 "

Decomposed at end, coming over dark. Sharp rise from 265 to 270°C with little coming over.

Our layout, as shown on Drawing So-2407 makes no provision for either general ventilation of the department or for the collection and further processing of the product discharged from the still at the end of the run. For general room ventilation we would suggest disc or propeller fans. For the collection of the fine dusts, decomposition products, etc., we would recommend hoods, duct-work, collector, etc., connected to an exhaust fan.

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The choice of a collector for this service is a difficult one. The material is acid and the fineness of particles approach that of fog particles. Wool cloth is quickly destroyed. To us it seems likely that the successful collector will be a scrub tower. The material caught in this tower would then be reworked to improve the yield.

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